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**A COMPARATIVE STUDY OF VERY HIGH BURNING RATE
MATERIALS - HIVELITE COMPOSITIONS 300511 AND 300435**

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AUGUST 1982



**US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
WEAPON SYSTEMS LABORATORY
DOVER, NEW JERSEY**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A series of safety and characterization tests were performed on a very high burning rate (VHBR) HIVELITE 300511 composition (Teledyne McCormick Selph product) in order to provide sufficient data so that a judgment can be made to qualify the material for in-service use. This composition is claimed to be less sensitive to electrostatics than HIVELITE 300435, a similar composition. The results of HIVELITE 300511 are compared to those for HIVELITE 300435. The range of tests include impact sensitivity, electrostatic sensitivity, friction		

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INTRODUCTION

The Naval Surface Weapons Center, Dahlgren Laboratory, is considering the use of a very high burning rate HIVEHITE material as a candidate for the rapid electric igniter primer, EX 164, for the 5"/54 guided projectile. Since the HIVEHITE composition selected was not qualified for in-service use, a series of safety and characterization tests was carried out by ARRADCOM at the request of the Naval Surface Weapons Center to determine the safety and classification data for the composition. The results of the study showed that HIVEHITE 300435 is very sensitive to electrostatic and friction stimuli and must be handled as a primary explosive (ref 1). HIVEHITE 300435, therefore, was modified to reduce the sensitivity hazards. The modified material is called HIVEHITE 300511.

OBJECTIVE

Although HIVEHITE 300511 was claimed to be identical but less sensitive than HIVEHITE 300435, it was deemed desirable to conduct the same series of safety and performance tests to ascertain that none of the other important properties had changed. The following report describes the results of such a study conducted on HIVEHITE 300511 by ARRADCOM at the request of the Naval Surface Weapons Center. A comparison is made with the data obtained on HIVEHITE 300435.

TEST PROGRAM AND RESULTS

The name HIVEHITE is a contraction of the words high velocity ignition. HIVEHITE is not a single formulation but is really a family of compounds based on salts of decaborane ($B_{10}H_{10}$) blended with a variety of oxidizers such as potassium nitrate. HIVEHITE is a trademark of Teledyne McCormick Selph (TMCS).

HIVEHITE 300435 is a fast burning pyrotechnic composition consisting of cesium boron hydride ($Cs_2B_{10}H_{10}$) with a potassium nitrate (KNO_3) oxidizer, a polyethylene glycol binder (7.5% by weight), and graphite (0.5% by weight). The formulation of HIVEHITE 300511 is $Cs_2B_{10}H_{10}/KNO_3/Wax/C: 18/75/6.5/0.5$ (ref 2). The difference between the two compositions is that the microcrystalline wax replaced the polyethylene glycol and the method of coprecipitation of the $Cs_2B_{10}H_{10}$ and KNO_3 was changed.

The program consisted of the following sensitivity and characterization tests which were performed in accordance with the requirements in Volume IV Joint Service Safety and Performance Manual for Qualification of Explosives for Military Use (Triservice Qualifications Manual, ref 3).

1. Impact sensitivity
2. Electrostatic sensitivity
3. Friction sensitivity

4. Vacuum thermal stability/compatibility
5. Differential thermal analysis (DTA)/thermogravimetric analysis (TGA) and differential scanning calorimetry.
6. Explosion Temperature
7. Autoignition
8. Density as a function of pressure
9. Effect of moisture
10. Physical stability
 - a. Linear coefficient of thermal expansion
 - b. Growth and exudation

In addition to the above, the following tests were conducted: Specific heat, burning rate, closed bomb, scanning electron microscopy (SEM), and the heat of explosion.

A description of the apparatus, the test procedures, and the results of the tests are listed below. Comparisons are made with HIVE-LITE 300435 and other common energetic materials tested under the same conditions.

IMPACT SENSITIVITY

The impact sensitivity tests were mainly performed on the ERL (NO.) Type 12 impact tester. The apparatus uses a 2.5-kg steel dropweight with the sample resting on sandpaper between two steel anvils.

A full firing curve, as well as the drop heights corresponding to the 50% and 10% probability of initiation, were obtained. The firing curve was determined by the run-down method, using 20 trials at each height. The 50% initiation point was determined by means of the Bruceton up-and-down method. The 10% value was the minimum height which resulted in initiation of the sample in at least one of 10 trials. The full run-down firing data for both HIVE-LITE compositions are listed in table 1. As can readily be seen, HIVE-LITE 300511 is less impact sensitive than HIVE-LITE 300435. Table 2 lists the 50% and the 10% firing points obtained by the Bruceton and the PA methods. Included are the 10% firing points obtained with the PA impact apparatus. (The PA impact tester utilizes a 2-kg steel drop weight with the sample in a confined environment.) For comparison purposes, the 50% and the 10% firing heights for other explosives are included. The data show that HIVE-LITE 300435 is more sensitive to impact than Comp B and RDX but is less sensitive than lead azide. HIVE-LITE 300511, on the other hand, is less sensitive than Comp B and RDX on the ERL tester, but is more sensitive than Comp B in the PA test, i.e., in a confined environment.

Teledyne McCormick Selph performed impact tests on both compositions with a 2-kg dropweight on a grit base. The 50% and 10% points for HIVEHITE 300435 were 30.5 cm and 12.0 cm respectively, while for HIVEHITE 300511 the maximum no-fire was 46 cm.

ELECTROSTATIC SENSITIVITY

The electrostatic sensitivity test was conducted on powder and pressed wafers of HIVEHITE 300511 using the approaching-electrode method. The pressed wafers were 0.64 cm (0.25 in.) in diameter and 0.048 to 0.051 cm (0.015 to 0.020 in.) thick. In addition, tests on HIVEHITE 300511 wafers were also conducted in a nitrogen atmosphere to determine whether the material can be handled safer in an inert atmosphere. This test was done by flushing the firing chamber with nitrogen for 5 minutes prior to test and flowing a stream of nitrogen over the wafer during the test.

The test results are compared to those of HIVEHITE 300435 in table 3. With powder samples of HIVEHITE 300435, initiation was obtained at 0.9 millijoules, the lowest value tested. With HIVEHITE 300511 powder, initiation occurred at 9 millijoules, but no initiation occurred in 20 trials at 6 millijoules. For comparative purposes, RD1333 lead azide has a minimum initiation energy of 5 to 6 millijoules and basic lead styphnate has less than 0.2 millijoule. The sensitivity of HIVEHITE to electrostatics places the material in the same category as most primary explosives. A value of 0.9 millijoule for HIVEHITE 300435 indicates that this material is electrostatically very sensitive, similar to basic lead styphnate. The 6 millijoule no-fire value for HIVEHITE 300511 in powder form indicates that it is as sensitive as RD1333 lead azide.

In the test conducted on wafers, the approaching electrode needle came to within 0.020 cm (0.008 in.) above the wafer. With HIVEHITE 300435 wafers, initiation was obtained at 1.1 millijoules, the lowest value tested. With HIVEHITE 300511 wafers, initiation occurred at 90 millijoules but no initiation occurred in 20 trials at 60 millijoules. The increase in energy by a factor of 10 from powder to pellet form is significant.

When conducted in the nitrogen atmosphere, similar results were obtained for the powder and pellet forms of HIVEHITE 300511.

Teledyne McCormick Selph performed an electrostatic sensitivity test on this material. The test was performed with a point-to-point configuration on powder in an open cup. Using a 500 picofarad capacitor and large heavy electrodes the initiation value for HIVEHITE 300511 was greater than 225 millijoules. With HIVEHITE 300435 an initiation value of 2.25 millijoules was determined (refs 1, 2).

FRICITION SENSITIVITY

The ARRADCOM (formerly called Picatinny Arsenal) large-scale friction pendulum apparatus used in this test consisted of a fixed steel anvil and a weighted pendulum with a steel and a fiber shoe. Both HIVEHITE 300435 and 300511 detonated with the steel and the fiber shoes. The test is normally run on main charge explosives.

Due to the results obtained on the ARRADCOM friction pendulum tester an additional test was conducted on HIVEHITE 300511 using the BAM friction apparatus for primary explosives. The BAM machine was built by Julius Peters of West Germany and consists of a roughened stationary porcelain rod and a moving roughened porcelain plate. The apparatus measures the friction sensitivity of a material in terms of a load in grams applied to the test material between the porcelain rod and plate. At a load of 2,075 grams, the maximum load of the apparatus, no reaction was obtained. For comparison purposes, basic lead styphate and RD1333 lead azide has a 10% initiation load of 40 grams.

The results obtained with the two friction apparatus indicate the HIVEHITE 300511 is less sensitive than most primary explosives and more or as sensitive as RDX and HMX.

Thermal Sensitivity

Differential Thermal Analysis/Thermogravimetric Analysis (DTA/TGA)

Simultaneous DTA/TGA (weight change measurement) were obtained for HIVEHITE 300511 with a Mettler TA-2 thermoanalyzer at a heating rate of 10 K/min in static air (figure 1). The DTA/TGA was also obtained in a nitrogen atmosphere (figure 2). In each atmosphere the tests conducted up to 773 K (500°C).

HIVEHITE 300511 DTA/TGA data are listed in table 4. Only one endotherm and four exotherms were noted. The endotherm with a peak temperature of 411 K (138°C) represents the alpha to beta phase transition of KNO_3 . This endotherm is followed by four exotherms with peaks at 538 K (265°C), 567 K (294°C), 598 K (325°C), and 653 K (380°C). During the first three exotherms, the sample lost 4.8% of its original weight, indicating that these reactions are essentially solid state. The weight loss during the final exotherm was 95.2%. The rapidity with which the weight loss and exothermal reaction occurred is characteristic of an ignition process. It should be noted that the endotherm due to the wax transition and melting was not detected with the Mettler.

HIVEHITE 300435 underwent two endothermal reactions followed by four exotherms (figure 3). The onset of the first endotherm occurs at 330 K (57°C) and peaks at 333 K (60°C) and is attributed to the melting of the polyethylene glycol (trade name Carbowax, manufactured by Union Carbide). The second endotherm, which starts at 403 K (130°C) and peaks at 408 K (135°C), results from the

crystalline transition of KNO_3 (alpha to beta). The onset of the first exotherm occurs at 453 K (180°C), and is accompanied by a weight loss indicating the commencement of the decomposition reaction. This initial exotherm is followed by three additional exothermic reactions with peaks at 543 K (270°C), 593 K (320°C), and 663 K (390°C). After an initial weight loss of 6% up to 653 K (380°C), the last exotherm is accomplished by a very rapid additional 60% weight loss which indicates an ignition reaction had occurred. The thermal events are listed in table 4.

The overall appearance of the two HIVELITES is similar: three exotherms follow the KNO_3 phase change during which approximately 5% weight loss occurred. Although the three exotherms occur sooner (at a lower temperature) for HIVELITE 300435, the final ignition exotherm peak occurs 10 K lower for HIVELITE 300511. HIVELITE 300511 lost all its weight while HIVELITE 300435 lost only 66% of its original weight.

The DTA thermal data for HIVELITE 300511 in a nitrogen atmosphere is shown in table 4. Two endotherms and two exotherms were observed. The first endotherm which peaked at 414 K (141°C) was the phase change of potassium nitrate. This was followed by a large endotherm which peaked at 567 K (294°C). This endotherm may be the melting of the coprecipitate. Following this endotherm, a small exotherm was observed which peaked at 582 K (309°C) and was accompanied by a very small weight loss. A highly exothermal reaction began at 595 K (322°C) which culminated in ignition at 666 K (393°C). During the ignition reaction the sample lost 88.4% of its weight.

A comparison of the experimental data obtained in air with that in nitrogen shows that HIVELITE 300511 is appreciably less stable in air than in nitrogen. It undergoes three highly exothermal reactions which is followed by ignition at a temperature 13 degrees lower than in nitrogen.

Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry experiments were conducted on samples sealed in aluminum containers and heated in the Perkin-Elmer DSC-2 in a nitrogen atmosphere.

Since the presence of the 6.5% wax additive was not detected in the DTA/TGA thermograms using the Mettler equipment, a relatively large sample (16 mg) was heated in the Perkin-Elmer DSC-2, which is a more sensitive apparatus. The results are listed in table 6.

A low temperature broad exotherm of small magnitude was observed over the temperature range 288 K (15°C) to 338 K (65°C) with a peak at 320 K (47°C). This broad endotherm ($\Delta T=50$ degrees) is attributed to the phase transition and melting of the wax. This was followed by two extremely large endotherms which bracketed a very small one. The first large exotherm is believed to be the transition of KNO_3 while the second large endotherm may result from the fusion of the coprecipitate. The peak temperatures of these reactions are 416 K (143°C) and 575 K (302°C) respectively. The peak of the small exotherm is 440 K (167°C). The ignition reaction began at 681 K (408°C) and peaked at 686 K (413°C).

In order to elucidate the endothermal reaction attributed to the fusion of the coprecipitate, a sample of HIVE-LITE 300511 was heated to 600 K (327°C) and then cooled at 10 K/min. A very large exotherm with onset and peak temperatures at 570 K (297°C) and 567 K (294°C) respectively, occurred. This is evidence of a reversible physical process which is probably the solidification of the coprecipitate. This is followed by the exotherm for the beta to alpha (β to α) phase transition of KNO_3 , and the phase change and solidification of the wax.

Vacuum Thermal Stability Test (VTS)

The 373 K (100°C) vacuum thermal stability test was conducted on a 5 g sample for 40 hours. The total amount of gas evolved after 40 hours was 3.56 mL and 0.36 mL for HIVE-LITE 300435 and 300511, respectively. The amount of gas evolved, 0.71 and 0.07 mL per g for 40 hours, is well below the maximum accepted value of 2.0 mL per g for 40 hours, and is termed as moderate.

Vacuum thermal stability tests were also conducted to determine the compatibility between HIVE-LITE 300511 and the potential contact interfaces in the HIVE-LITE RIP primer. The materials tested were M30 propellant, nitrocellulose (12.6% N), cellulose acetate butyrate (CAB), Velostat, and epoxy adhesive, EC 2216. Negligible gas was evolved. The tests were conducted at 373 K (100°C) for CAB, Velostat and the epoxy adhesive, EC 2216, and at 363 K (90°C) for the two propellants, M30 and NC. The results are listed in table 7.

Explosion Temperature

The explosion temperature test was conducted by immersing a copper blasting cap containing approximately 40 milligrams of sample in a confined state to a fixed depth into a molten Wood's metal bath. Time to explosion was determined by measuring the time required for the blasting cap to rupture. The temperature of the 5 second point is usually reported. For HIVE-LITE 300435 the 5 second point was 776 K (503°C). The 1 second point was 883 K (610°C). A 5-second temperature was not obtained for HIVE-LITE 300511 because the furnace burned out during the test. Data obtained before the furnace broke, however, showed that the 5-second temperature was greater than 773 K (500°C). The TMCs reported a 5-second temperature of 779 K (506°C).

Autoignition Temperature

The autoignition temperature was determined by a method using DTA (ref 4). This method utilizes several heating rates and their respective onset and peak exotherm temperatures to solve the Kissinger's equation (1).

$$k = \frac{E\phi}{RT^2} = Ae^{-\frac{E}{RT}} \quad (1)$$

where E = apparent activation energy (cal mole⁻¹)

k = rate constant (min⁻¹)

A = frequency factor (sec⁻¹)

R = gas constant (1.987 cal K⁻¹ mole⁻¹)

T = peak exotherm temperature (K)

ϕ = heating rate (K min⁻¹)

The autoignition temperature was obtained by extrapolating the data to a near zero heating rate assuming a rate constant of 0.05 min⁻¹.

Two different DTA techniques were used in this study. The first method used a Perkin-Elmer DSC-2 at four heating rates, 5, 10, 20, and 40 degrees per minute. The samples were confined. In the second method, the samples were heated unconfined in a nitrogen atmosphere at five heating rates (1.3, 2.6, 5.2, 10.5, and 21 degrees per minute) using a Deltatherm III thermoanalyzer. The onset and peak exotherm temperatures and the respective heating rates are summarized in table 8 respectively. The apparent activation energy, frequency factor, and the autoignition temperature for the confined method were 39 kcal/mole, 2.0×10^{10} sec⁻¹, and 635 K (362°C), respectively. The values for the unconfined method were 41 kcal/mole, 4.4×10^{12} sec⁻¹, and 633 K (370°C).

Density

The density of 1.27 cm (0.50 in.) long pellets was determined as a function of pressure at four different pressures. HIVEHITE 300435 was pressed under vacuum with a 60 second dwell time. The diameter of the pellets was 1.895 cm (0.75 in.). In this present study HIVEHITE 300511 was pressed at atmospheric pressure with a 15 second dwell time. The pellets had a 1.27 cm (0.50 in.) diameter. The results are listed in table 6. The average particle size for the material ranges from 20 to 40 microns, the theoretical density is 2.11 g/cm³ and the bulk density is 0.80 g/cm³.

Effect of Moisture

To determine the effect of exposure to moisture, pressed HIVEHITE pellets were exposed to ambient air for 7 days and to a very high relative humidity atmosphere (90 to 99%) for 7 days. The high humidity environment was obtained by placing the pellets on an aluminum dish above water while in a dessicator.

Two types of HIVEHITE 300435 pellets were tested previously; black graphited pellets from TMcS lot No. 2 and white pellets from TMcS lot No. 5. The pellets were 0.61 cm (0.24 in.) diameter by 0.61 cm (0.24 in.) long with a den-

sity of 1.69 g/cm^3 . In this present study, three groups of HIVEHITE 300511 graphited pellets (lot No. 103) were tested; one group was supplied by TMCS and two groups were prepared at ARRADCOM. The TMCS pellets were 0.70 cm (0.275 in.) in diameter and 1.27 cm (0.50 in.) long with a center hole about 0.32 cm (0.125 in.) in diameter. The pellets prepared at ARRADCOM were 1.27 cm (0.50 in.) in diameter by 1.27 cm (0.50 in.) long. The only difference between these two groups was the density: one group was 1.54 g/cm^3 and the other was 1.92 g/cm^3 .

The results are shown in table 10. In the previous ambient air test with HIVEHITE 300435, the black graphited pellets (lot No. 2) had an average weight loss of 0.25% and an average decrease in overall length of 4.8%. The white pellets (lot No. 5) had an average weight loss of 0.19% and an average length decrease of 0.83%. No change was noted in the diameter of either lot. In the present study, only negligible dimensional and weight change were obtained with the three groups of HIVEHITE 300511 pellets.

In the previous very high humidity test, it was noted that a small amount of water had accumulated in the aluminum dish with the pellets made from HIVEHITE 300435. These pellets were soft and distorted; the black pellets from lot No. 2 had changed color to almost white and had an average weight loss of 18%. The white pellets (lot No. 5) had an average weight loss of 10%. In the present study, no water accumulated in the aluminum dish. The HIVEHITE 300511 pellets were still black and not distorted. However, the pellets were soft. The pellets supplied by TMCS were too soft to measure and had an average gain of 17%. The 1.54 g/cm^3 density pellets had a 12.5% weight gain and an average increase in diameter and overall length of 0.8%. The 1.92 g/cm^3 density pellets had a 11% weight gain, an average increase in diameter of 3% and an average increase length of 9%.

The results indicate that HIVEHITE 300511 is less hygroscopic than HIVEHITE 300435. However, under the high humidity conditions, HIVEHITE 300435 and 300511 are both hygroscopic. At which level the performance of the material would be affected is not known.

Physical Stability

The HIVEHITE 300511 was subjected to two different tests to determine whether it could maintain its integrity throughout the normal temperature range. The first test was the determination of the linear coefficient of thermal expansion; the second test determined any growth or exudation characteristics due to temperature cycling.

Linear Coefficient of Thermal Expansion

In the previous study, the linear coefficient of thermal expansion was obtained for two groups of HIVEHITE 300435 pellets. In this study, three groups of HIVEHITE 300511 pellets were tested. One group of HIVEHITE 300435 pellets and two groups of HIVEHITE 300511 pellets were prepared at ARRADCOM. The other

groups were supplied by TMCs. The pellets prepared at ARRADCOM were 0.635 cm (0.25 in.) in diameter and 0.635 cm (0.25 in.) long. The HIVEHITE 300435 pellets supplied by TMCs were 0.61 cm (0.24 in.) in diameter and 0.61 cm (0.24 in.) high with a axial hole 0.18 cm (0.070 in.) in diameter. The HIVEHITE 300511 pellets were 0.70 cm (0.275 in.) in diameter and 1.09 cm (0.43 in.) long with an axial hole almost 0.32 cm (0.125 in.) in diameter.

The linear coefficient of thermal expansion of the pellets were determined using a Perkin-Elmer Thermomechanical Analyzer, Model TMS-1. Each pellet was heated at 5 K/min from -213 to 353 K (-60 to 80°C). The results are summarized in table 11. Slightly different results were obtained for the different groups of pellets. The linear coefficient of thermal expansion was linear throughout the entire temperature range only for the TMCs holed pellets of HIVEHITE 300435 and the 1.55 g/cm³ density pellets of HIVEHITE 300511. The other groups exhibited two different linear regions over the same temperature range. It can also be seen from the table that over the wide temperature range of approximately 213 to 313 K (-60 to 40°C), the expansion coefficient of the three HIVEHITE 300511 groups are nearly identical, approximately $63 \times 10^{-6}/K$.

Specific Heat

The specific heat was determined for HIVEHITE 300511 by heating the sample, a sapphire disc of known weight, and an empty pan in a Perkin-Elmer DSC-2 from 240 K (-23°C) to 345 K (71°C). The displacement of sample, empty pan, and sapphire were measured at various selected temperatures, and the specific heat was calculated using the following equation:

$$C_{p \text{ sample}} = \frac{W_{\text{sapphire}}}{W_{\text{sample}}} \times \frac{D_{\text{sample}}}{D_{\text{sapphire}}} \times C_{p \text{ sapphire}} \quad (2)$$

where

C_p = heat capacity (cal g⁻¹ K⁻¹)

W = weight

D = displacement (cm)

The specific heat values for a number of different temperatures are given in table 12. A discontinuity appeared in the specific heat curve for the sample due to the wax transition and fusion which causes the specific heat measurement to increase and then decrease.

Growth and Exudation

The growth characteristics of HIVEHITE were determined by temperature cycling pressed pellets, between 219 K (-54°C) (-65F) and 333 K (60°C) (140F) for 30 cycles. In the previous test, the HIVEHITE 300435 pellets were 1.90 cm (0.75

in.) in diameter and 1.27 cm (0.50 in.) long. In the present test, the HIVEHITE 300511 pellets were 1.27 cm (0.50 in.) in diameter by 1.27 cm (0.50 in.) long. As can be seen in table 13, the irreversible volume changes were 0.93% for HIVEHITE 300435 pellets having a density of 1.94 g/cm³ and 1.00% and 1.13% for HIVEHITE 300511 pellets having densities 1.57 and 1.83 g/cm³ respectively. The maximum permissible volume is 1.0%.

For the exudation test, two HIVEHITE cylinders were clamped together between steel plates to an initial pressure of 0.414 MPa (60 psi). The clamped ensemble was then subjected to 30 cycles from ambient to 333 K. No exudation was noted after the test.

Small Scale Gap Test

The small scale gap test without a gap was used to evaluate the shock sensitivity of HIVEHITE 300511. In this test, the standard donor explosive (Composition A-5) and the test material were pressed into identical thick wall brass cylinders, 2.54 cm (1.0 in.) o.d., by 0.5 cm (0.2 in.) i.d., by 3.8 cm (1 1/2 in.) long. The donor was placed on top of test material and initiated with an electric detonator. The donor provided an explosive shock to the test material. HIVEHITE 300511 did not detonate in the diameter and length noted, showing that it is not shock sensitive in the confined configuration tested.

Scanning Electron Microscopy and Hot State Polarizing Microscopy Examination

In an effort to determine whether the wax melted in the HIVEHITE 300511 composition, scanning electron microscopic (SEM) photographs were taken of the composition at ambient temperature and at 473 K (200°C). The SEM photos were taken with a magnification of 500 X and 300 X (figs. 4 and 5).

Since close examination of the SEM photos did not reveal any significant change in the composition, a decision was made to examine the material with a hotstage polarizing microscope.

A sample of HIVEHITE 300511 was heated at 10 K/min to 473 K. No change was noted. It was cooled at 3 K/min to ambient, again with no change. The sample was reheated at 10 K/min and at 315.5 to 318.7 K the particles shifted position. This may have been caused by a crystallographic change (polymorphic change) with dimensional/structural change with one of the components. At about 478 K the window of the hotstage fogged considerably, indicating sublimation and/or decomposition. Tiny, well-formed crystals appearing in various bright colors under polarized light, seemed to be unaffected by heating up to 573.5 K. At this temperature the sublimation and/or decomposition ceased. The magnification of the hotstage polarizing microscope is 105 X.

Burning Rate Measurements

A modified version of the TMcS standard primer acceptance test was used to determine the burn rate measurement of HIVE LITE 300511 and to reproduce the TMcS burn rate values for the material. Also an effort was made to determine the effect of burning rate versus pressed density of the same material.

In the modified TMcS procedure perforated pellets 0.635 cm in diameter X 1.25 cm long (1.25 in. dia. X 0.5 in. long) with a 0.18 cm (0.070 in.) center hole and a density of 1.57 g/cm³ were stacked to a length of 15.24 cm (6 in.) within a plastic tube. The plastic flash tube was notched at 10.51 cm (0.20 in.) intervals with a "V" cut, leaving a 0.076 cm (0.030 in.) wall thickness at the bottom of each notch. These notches assure uniform fragmentation of the flash tube as burning of the pellets progressed within the tube. Also six lead fuse timing wires were placed at the base of a notch at 2.85 cm (1.200 in.) interval. Each of the wires short out in series of resistors. The tubes were obtained from TMcS.

The stack of pellets in the flash tube was initiated by 0.25 g of Class 7 black powder, which was activated by an electric match. As each of the lead fuse timing wires progressively break, the circuit resistance changes and the associated voltage drop is timed and recorded with a Nicolet, Explorer III. Results were recorded in only one of the three tests conducted. The results are listed in table 14. The average burning progression rate by this method was 954 m/s. The TMcS data using this method ranged from 1220 to 1325 m/s (ref 2).

Additional tests were conducted with HIVE LITE 300511 (Lot 103) when pressed into solid cylindrical pellets, 1.25 cm. dia X 1.25 cm long at two densities, 1.55 and 1.89 g/cm³. Surprisingly these pellets burned very slowly and their progression rates could not be measured by the above technique. The flash tube holder would not rupture uniformly in relation to the burning within the tube because of the slow rate at which the pressure was developing.

Therefore, the ends of the pellets were covered with tape and then dipped in hot black asphalt paint for inhibition in order to control the direction of burning. Three coats were applied to the cylindrical surface in this manner. Four pellets were stacked on a wooden base with 1/4 amp lead fuse wire placed between each pellet to time the burning of each segment. The portion of fuse wire in contact with the pellet was rolled flat to less than 1 mm. The entire assembly was held together and the fuse wires protected with soft modeling clay. The top pellet was initiated and the timing measurements made as previously described. The results are listed in table 15.

The results by this method show a drastic difference with the results obtained with the flash tube. With a density of 1.55 g/cm³ the burning rate was 8.09 cm/s and with a density of 1.99 g/cm³ the burning rate reduced to 3.34 cm/s.

It is to be noted that the burning rate versus density measurements are reported in cm/s units as compared to the flash tube progression rate which is reported in meters/s. The tremendous difference in rates (a factor of 11,800 to 28,000 depending on density) indicates that the surface area initiation is the

predominant parameter being measured in the flash tube. The surface area is rapidly initiated and based on the burning rate data, burning penetrates to an insignificant depth into the body of the pellet in the time available. The bulk of the material is left to burn in space as the flash tube shatters away from the burning propellant.

The term burning rate implies a linear regression of the surface and is a misnomer when applied to uninhibited flash tube burning. The measurements include a rate of surface area initiation simultaneously with normal linear regression rates. The strength of the flash tube holder is a factor and will effect the time of rupture in relation to the quantity of material being burned.

The data also shows that there is a considerable burning rate versus density effect. In addition the segmented measurements indicate that the burning rate does not accelerate over the 2 inch length burned at atmospheric pressure.

A similar test with HIVEHITE 300435 pellets with a density of 1.94 g/cm^3 produced a burning rate of 418 m/s (ref 1).

The fact that the replacement of the polyethylene glycol binder in HIVEHITE 300435 to wax in HIVEHITE 300511 (along with the coprecipitator method) can reduce the apparent burning rate by a factor of thousands suggests that the wax acts as an internal inhibitor which strongly interferes with the internal ignition propagation mechanism.

A significant observational difference was noted during the testing of HIVEHITE 300435 and 300511. HIVEHITE 300435 burns violently and with a very loud noise that could be mistaken for a detonation. HIVEHITE 300511 burns with a hissing noise similar to the open air burning of black powder. The soft modeling clay used to hold together the stacked assembly of pellets survived the reaction leaving a hollow mold of the pellets.

Closed Bomb Testing

Pressed pellets, 0.96 cm (0.378 in.) length and 0.64 cm (0.253 in.) diameter, were used for the closed bomb tests. These pellets were pressed to a density of 1.60 g/cm^3 which is approximately 75% of theoretical maximum density (TMD). The combustion characteristics were determined for approximately 0.05 loading density in a 200 cm^3 closed bomb at sample temperatures of 294 K (+21°C) and 224 K (-51°C). HIVEHITE pellets were packaged in a plastic bag and ignited by a squib igniter with 0.2 g of black powder. Pressure time data were recorded on a Nicolet Digital Oscilloscope and subsequently reduced by standard methods. Figures 6 and 7 show typical pressure time data at ambient and cold temperatures. Three shots were fired at ambient and two at cold temperature. The dp/dt versus p curves derived from smoothed pressure time data are shown in figures 8 and 9.

Burning rate parameters were determined from composite data at the two temperatures. Least squares fits were made to the AP^B and $AP^B + C$ burning rate equations. These fits are shown in figures 10 and 11.

A 19 point smoothing procedure was used to condition the pressure-time data before the dp/dt data were derived. It is obvious that the combustion behavior is erratic and that the apparent burning rates calculated probably do not correspond to linear surface regression rates. The calculations do indicate lower rates at the lower conditioning temperature which in turn indicates similar burning behavior at cold and ambient temperatures.

Burn rate data was obtained by TMCS on HIVEHITE 300511 (ref 2). The TMCS data was obtained at higher loading density (0.18 g/cm^3) and at ambient temperature with samples compressed to 75% TMD (1.56 g/cm^3). The pellets were loaded in metal cups and the burn rate data was obtained assuming a cigarette type burn with the outer surface inhibited. This data is shown in figure 12.

Comparisons between data of this study and the TMCS data can be made in the (200 to 2000 psi range) for 75% TMD at ambient temperature. Table 16 shows the burn rate parameters determined from the data of this program and that from TMCS. Calculating the predicted burn rates at 6.9 MPa (1000 psi), values of 79.25 and 71.88 cm/s (31.2 and 28.3 in/s) were obtained from the two fits to the ARRADCOM data and 5,058.4 cm/s (199.5 in/s) from the fit to the TMCS data.

The wide disparity in these apparent burn rates must reflect the effects due to confinement in the TMCS method of determining the burn rate. More careful comparisons and controlled experiments are indicated to establish the true burning rate for HIVEHITE 300511. Table 16 indicates comparable data for HIVEHITE 300435.

Additional Data

Table 17 lists several specific thermochemical properties determined by TMCS (ref 2). In this study the heat of explosion was obtained experimentally. In two runs the values obtained were 988 and 987.8 cal/g. In table 17, TMCS reports a value of 750 cal/g.

SUMMARY AND CONCLUSIONS

A series of safety and characterization tests were performed on HIVEHITE 300511 composition in order to provide sufficient data so that a judgment can be made to determine whether or not the material is safe to handle and to qualify the material for in-service use. The tests conducted were impact sensitivity, electrostatic sensitivity, friction sensitivity, shock sensitivity, explosion temperature, autoignition vacuum thermal stability, DTA/TGA, loading density, linear coefficient of thermal expansion, specific heat, growth and exudation, the effect of moisture, burning rate tests and closed bomb tests. Comparisons were made to similar data obtained for HIVEHITE 300435.

The ERL impact test results indicate that HIVEHITE 300511 is much less sensitive to impact than HIVEHITE 300435 and Comp B. With the PA impact tester the PA 10% point indicates that in that confined state HIVEHITE 300511 is almost as sensitive as RDX.

HIVELITE 300511 is still sensitive to friction and electrostatic stimuli and should be handled as a primary explosive. Electrostatically it appears to be less sensitive than HIVELITE 300435 and lead styphnate but as sensitive as RD1333 lead azide. From powder to pellet form the no-fire value went from 6 to 60 millijoules, a factor of 10. The BAM friction apparatus for primary explosives indicated that HIVELITE 300511 is less sensitive to friction than lead styphnate and RD1333 lead azide.

Burning rate measurements were conducted in air under different conditions. Using the modified TMcS method 0.635 cm diameter perforated pellets stacked 15.25 cm in a plastic tube produced a progression rate of 954 m/s. However, solid HIVELITE 300511 pellets 1.27 cm dia X 1.25 cm long stacked four high which were dipped in hot black asphalt paint for inhibition to control the direction of burning produced surprisingly much lower burning rates. For a density of 1.55 g/cm³ the burning rate was 8.09 cm/s and for 1.89 g/cm³ the burning rate reduced to 3.34 cm/s.

In closed bomb tests HIVELITE 300511 pellets 0.96 cm dia and 0.64 dia were fired at ambient and cold temperatures. Values of 79.25 and 71.88 cm/s were obtained. However, TMcS data with HIVELITE 300511 loaded in metal caps produced a value of 5,058.4 cm/s. The wide disparity in these results may be due to the effects of confinement.

On an overall basis, HIVELITE 300511 is not as sensitive to impact, friction, and electrostatics as HIVELITE 300435; however, since HIVELITE 300511 is as sensitive to electrostatics as RD1333 lead azide it also should be handled as a primary explosive and proper precautions should be taken. HIVELITE 300511 is less hygroscopic than HIVELITE 300435. However, steps should be taken to prevent the presence of moisture in any application of HIVELITE 300511. Sufficient data has been obtained to evaluate HIVELITE 300511 for interim qualification for in-service use.

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3. Joint Services Evaluation Plan for Preferred and Alternate Explosive Fills for Principal Munitions, Volume IV, Joint Service Safety and Performance Manual for Qualification of Explosives for Military Use (Based on OD-44811), 12 May 1972, (AD-A086259).
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Table 1. Impact sensitivity run-down data

(ERL-Type 12 Tool, 2 1/2-kg Drop Weight)

<u>Ht</u> <u>(cm)</u>	<u>HIVELITE</u> <u>300435</u> <u>(% fired)</u>	<u>HIVELITE</u> <u>300511</u> <u>(% fired)</u>
20	5	-
30	35	-
40	45	5
60	85	25
70	100	25
100		45
150		65
240		75

Table 2. The 50% and 10% impact sensitivity data

<u>Explosive</u>	<u>ERL 50%</u> <u>firing ht (cm)</u>	<u>ERL 10%</u> <u>firing ht (cm)</u>	<u>PA 10%</u> <u>firing ht (in.)</u>
HIVELITE 300435	36.1 ± 2.1	19	6
HIVELITE 300511	129 ± 3.2	45	9
Comp B	60	35	14
RDX	39	31	8
RD 1333 lead azide	4	-	3

Table 3. Electrostatic sensitivity data

<u>Explosive</u>	<u>Minimum initiation energy</u> <u>(millijoules)</u>
HIVELITE 300435	> 0.9
HIVELITE 300435 wafer	> 1.1
HIVELITE 300511	6
HIVELITE 300511 wafer	60
Basic lead styphnate	> 0.2
RD 1333 lead azide	6

Table 4. DTA results for HIVEHITE 300511

a. Heating rate: 10K/min in static air

<u>Type of event</u>	<u>Onset</u>		<u>Peak</u>		<u>Comment</u>
	<u>K</u>	<u>°C</u>	<u>K</u>	<u>°C</u>	
endotherm	405	132	411	138	KNO ₃ phase change
exotherm	525	252	538	265	decomposition
exotherm	561	288	567	294	decomposition
exotherm	575	302	598	325	decomposition
exotherm	636	363	653	380	ignition

b. Heating rate: 10K/min in nitrogen atmosphere

endotherm	408	135	414	141	KNO ₃ phase change
endotherm	553	280	567	294	fusion of coprecipitate
exotherm	567	294	582	309	slight decomposition
exotherm	595	322	666	393	ignition

Table 5. DTA results for HIVE LITE 300435

Heating rate: 10 K/min in static air

Type of event	Onset		Peak		Comment
	K	°C	K	°C	
endotherm	330	57	333	60	M.P. of polyethylene glycol
endotherm	403	130	408	135	KNO ₃ phase change
exotherm	453	180	473	200	decomposition
exotherm	513	240	543	270	decomposition
exotherm	567	294	593	320	decomposition
exotherm	607	334	663	390	ignition

Table 6. DSC results for HIVE LITE 300511

Heating rate: 10K/min in sealed container in nitrogen atmosphere

Type of event	Onset		Peak		Comment
	K	°C	K	°C	
endotherm	288	15	320	47	wax transition and melting
endotherm	405	132	416	143	KO ₃ phase change
endotherm	438	165	440	167	
endotherm	558	285	575	302	may be fusion of coprecipitate
exotherm	681	408	686	413	ignition

*This exotherm was obtained from another experiment using a very small sample to avoid damage to the Perkin-Elmer DSC-2 apparatus.

Table 7. Vacuum stability test results

Results Sample Designation	100°C vacuum stability	
	<u>mL gas</u>	<u>hours</u>
HIVELITE 300511	0.18	40
CAB	0.07	40
Mixture	0.08	40
Difference	-0.17 ^a	40
HIVELITE	0.18	40
Velostat	0.25	40
Mixture	0.30	40
Difference	-0.13 ^a	40
HIVELITE	0.18	40
Epoxy EC 2216	0.32	40
Mixture	0.08	40
Difference	-0.42 ^a	40
Results Sample Designation	90°C vacuum stability	
	<u>mL gas</u>	<u>hours</u>
HIVELITE 300511	0.36	40
M30 Prop	1.10	40
Mixture	1.13	40
Difference	-0.33 ^a	40
HIVELITE	0.36	40
NC (12.6% N)	0.39	40
Mixture	0.37	40
Difference	-0.38 ^a	40

^aAmount of gas evolved by mixture in comparison with that evolved by materials individually indicates a negligible reactivity. Procedure PATR 3278 Rev 1.

Table 8. Calculation of activation energy, frequency factor, and autoignition temperature for HIVE LITE 300511

a. Perkin-Elmer DSC-2 Results

ϕ K/min	Onset temp K	Peak temp K	$1/T \times 10^{-3}$ (AK) ⁻¹	$\log \frac{\phi}{T^2}$
5	667.8	668.2	1.4966	-4.9508
10	684.0	685.5	1.4588	-4.6720
20	698.0	699.0	1.4306	-4.3880
40	713.5	716.2	1.3963	-4.1080

y intercept = 7.7883

m, slope = -8521.287

correlation coefficient = -0.9984

E = 38,993 kcal/mole (Activation Energy)

A = $2.02 \times 10^{10} \text{ sec}^{-1}$ (Frequency Factor) (Standard Mean Deviation = $0.047 \times 10^{-10} \text{ sec}^{-1}$)

T_i = (362C) 635K (Autoignition Temperature)

b. Deltatherm III Thermoanalyzer Results

ϕ K/min	Onset temp K	Peak temp K
1.3	645.5	649
2.6	659.3	661.3
5.2	675.5	678
10.5	687.3	685.8
21.0	703.4	706.6

E = 41.2 kcal/mole (185 joules/mole)

A = $4.8 \times 10^{12} \text{ sec}^{-1}$

T_i = (370°C) 643 K

Table 9. Loading density as a function of pressure

<u>Pressure</u>		<u>Hivelite 300435^a</u> <u>density (g/cm³)</u>	<u>Hivelite 300511^b</u> <u>density (g/cm³)</u>
<u>MPa</u>	<u>psi</u>		
34.5	5,000	1.61	1.60
69.0	10,000	1.88	1.79
103.4	15,000	1.89	1.82
137.9	20,000	1.94	1.94

^a1.895 cm (0.75 in.) diameter by 1.27 cm (0.50 in.) long pellets pressed under vacuum with a 60 second dwell time.

^b1.27 cm (0.05 in.) diameter by 1.27 cm (0.50 in.) long pellets pressed at atmospheric pressure with a 15 second dwell time.

Table 10. Effect of moisture on HIVELITE pellets

Ambient Air for Seven Days

<u>HIVELITE</u>	<u>Description</u>	<u>Density (g/cm³)</u>	<u>dia.</u>	<u>% Change length</u>	<u>wt</u>
300435 TMcS lot #2	black pellets, 0.24" dia. X 0.24" long	1.69	0	4.8	-0.25
300435 TMcS lot #5	white pellets, 0.24" dia. X 0.24" long	1.69	0	0.18	-0.19
300511 TMcS lot #103	black pellets with center hole 0.275" dia. X 0.5" long	-	0	0	0
300511 ARRADCOM	black pellets, 0.5" dia. X 0.5" long	1.54	0	0	0
300511 ARRADCOM	black pellets, 0.5" dia. X 0.5" long	1.92	0	0	0

High Humidity^A for Seven Days

300435 TMcS lot #2	black pellets, 0.24" dia. X 0.24" long	1.69	B	B	-18
300435 TMcS lot #5	white pellets, 0.24" dia. X 0.24" long	1.69	C	C	-10
300511 TMcS lot #103	black pellets with center hole, 0.275" dia. X 0.5" long	-	D	D	17
300511 ARRADCOM	black pellets, 0.5" dia. X 0.5" long	1.54	0.8	0.8	12.5
300511 ARRADCOM	black pellets, 0.5" dia. X 0.5" long	1.92	3.3	9	11

A: Pellets on aluminum dish above water in dessicator.

B: Pellets distorted, black pellets turned white.

C: Pellets distorted.

D: Pellets too soft to measure.

Table 11. Thermal expansion data

HIVELITE 300435

a. Pellets made at ARRADCOM - (0.635 cm dia by 0.635 cm high)

<u>Linear Coefficient of expansion X 10⁶/K</u>	<u>Temp range</u>		<u>Density g/cm³</u>
	<u>K</u>	<u>C</u>	
53	213 to 297	-60 to 24	1.93
66	298 to 323	25 to 50	

b. TMcS Lot No. 3 (with center hole)

47	213 to 353	-60 to 80	1.60
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HIVELITE 300511

a. Pellets made at ARRADCOM - (0.635 cm dia by 0.635 cm high)

64	213 to 349	-60 to 76	1.55
63	217 to 327	-56 to 54	1.67
75	327 to 353	54 to 80	1.67

b. TMcS Lot No. 103 (with center hole)

62	221 to 313	-52 to 40	1.57
70	313 to 353	40 to 80	1.57

Table 12. Specific heat of HIVEHITE 300511

Temperature		Specific heat cal g ⁻¹ deg ⁻¹
K	°C	
253	-20	0.22
273	0	0.23
298	25	0.24*
313	40	0.28*
322	49	0.32*
333	60	0.28
344	71	0.28

*The specific heat values include the heat absorbed for wax transition and fusion

Table 13. HIVEHITE growth qualification test results

	<u>300435</u>	<u>300511</u>			
initial density (g/cm ³)	1.94	1.57	1.60	1.81	1.93
initial dia. (cm)	1.90	1.27	1.27	1.27	1.27
initial length (cm)	1.25	1.30	1.23	1.52	1.40
wt. change (%)	0.21	0.03	0.01	0.01	-0.44
density change (%)	0.78	0.98	1.00	1.12	2.06
volume change (%)	-0.93	-0.99	-1.01	-1.12	-2.44

Table 14. HIVE LITE 300511 progression rate at atmospheric pressure

Pellets: 0.625 cm dia X 1.27 cm long in 15.24 cm stack in flash tube

Density: 1.57 g/cm³

<u>Break wire no.</u>	<u>Interval</u>		<u>Δt (μs)</u>	<u>Progression rate (m/s)</u>
	<u>cm</u>	<u>(in.)</u>		
1 - 2	2.85	1.200	5.95	*5123
2 - 3	2.85	1.200	33.35	914
3 - 4	2.85	1.200	33.95	898
4 - 5	2.85	1.200	28.50	1069
2 - 5	8.55	3.600	95.80	954

*Omitted

Table 15. HIVELITE 300511 Lot 103

Burning rate versus density

Pellets: 1.25 cm dia x 1.27 cm long

a. Density = 1.55 g/cm³

<u>Break wire no.</u>	<u>Test no. 5</u>	<u>Test no. 6</u>	<u>Test no. 7</u>	<u>Test no. 8</u>	<u>Average</u>
	cm/s	cm/s	cm/s	cm/s	
1 - 2	8.08	7.03	9.09	-	
2 - 3	6.46	7.53	7.20	6.78	
3 - 4	9.04	7.82	-	15.92	
4 - 5	9.45	6.92	9.56	7.85	
1 - 5	8.08	7.31	8.73	8.25	8.09 cm/s

b. Density = 1.89 g/cm³

	<u>Test no. 5</u>	<u>Test no. 4</u>	
1 - 2	4.19	4.45	
2 - 3	2.29	4.43	
3 - 4	4.36	3.90	
4 - 5	3.81	1.89	
1 - 5	3.43	3.24	3.34 cm/s

Table 16. HIVELITE 300511 closed bomb burn rate parameters (in./s)

at $\rho = 1.56 \text{ g/cm}^3$ and 294 K (21C)

$$r_B = 0.009P^{1.18} \quad (150 < P < 1750)$$

$$r_B = 0.00079P^{1.83} + 3.9$$

TMcS data produces for almost same conditions:

$$r_B = 0.012P^{1.74} \quad (200 < P < 2000)$$

at $\rho = 1.56 \text{ g/cm}^3$ and 222 K (-51C)

$$r_B = 0.0114P^{1.10} \quad r_B = 0.00013P^{1.71} \quad (150 < P < 1750)$$

For HIVELITE 300435, TMcS data shows that at $\rho = 1.64 \text{ g/cm}^3$ at ambient conditions.

$$r_B = 0.0116P^{1.64} \quad (1000 < P < 2000)$$

$$r_B = 5.58P^{-0.74} \quad (0 < P < 1000)$$

Table 17. HIVELITE 300511 thermochemical properties

Impetus (ft-lb/lb)	159,000
I_{sp} (sec)	198
Heat of explosion (cal/g)	750
T_p (K)	2283
Gamma	1.151
Product molecular weight	46.0
Gas molecular weight	44.0
Moles product/100 g	2.17
Moles gas/100 g	1.97
Theoretical max density g/cm ³	2.074

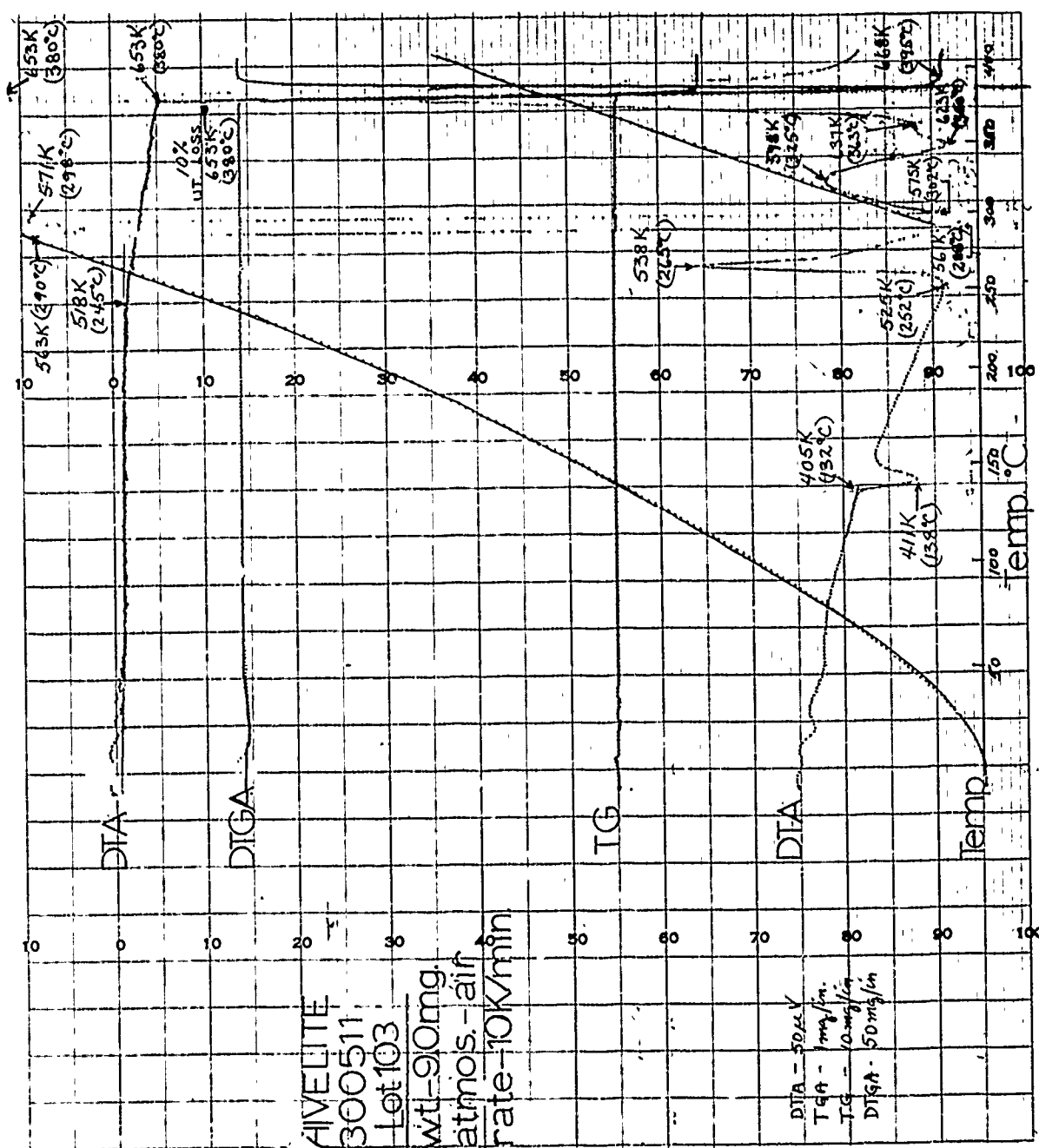


Figure 1. DTA/TGA thermograms of HIVELITE 300511 in air

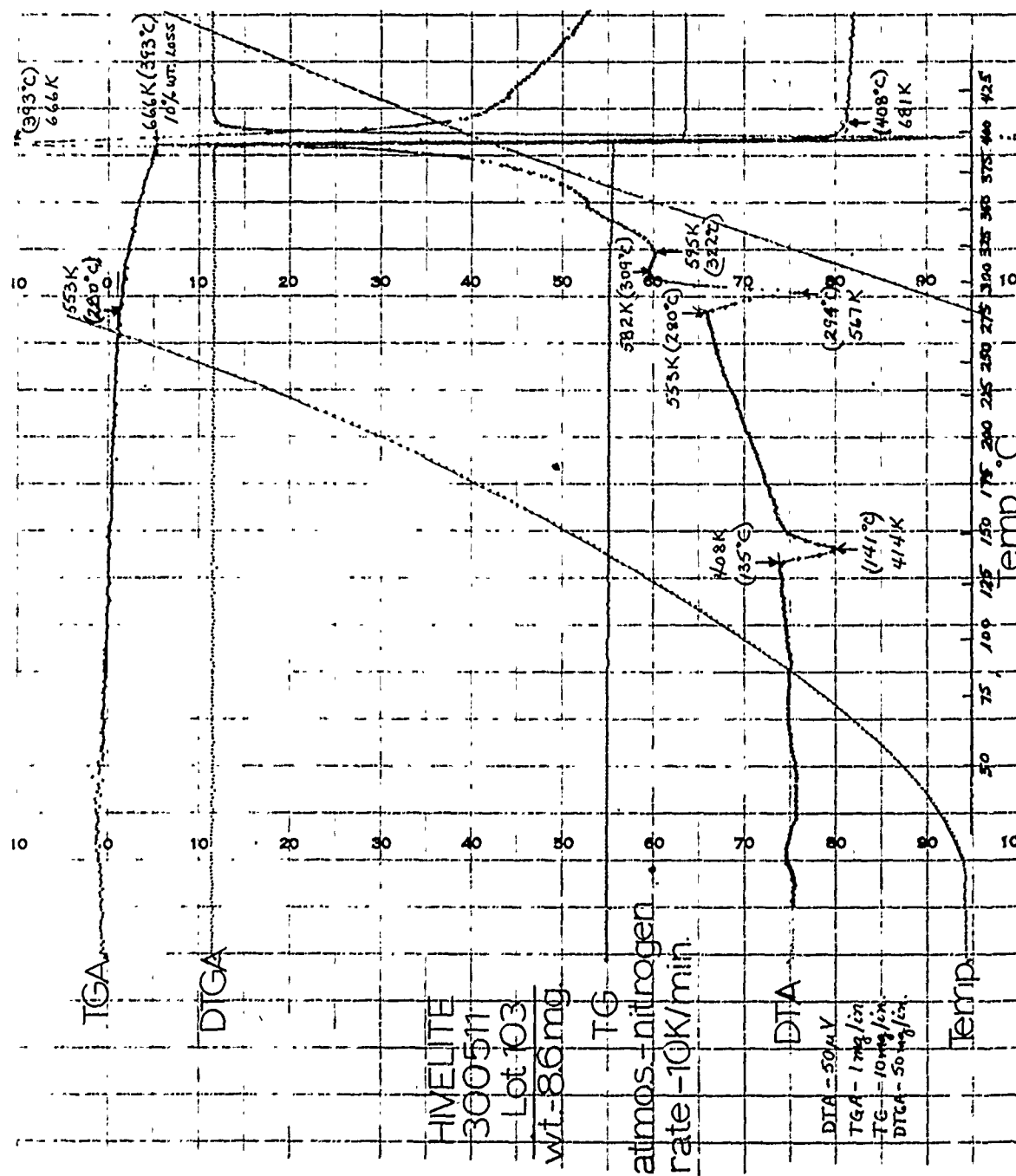


Figure 2. DIA/TGA thermograms of HIVE LITE 300511 in nitrogen

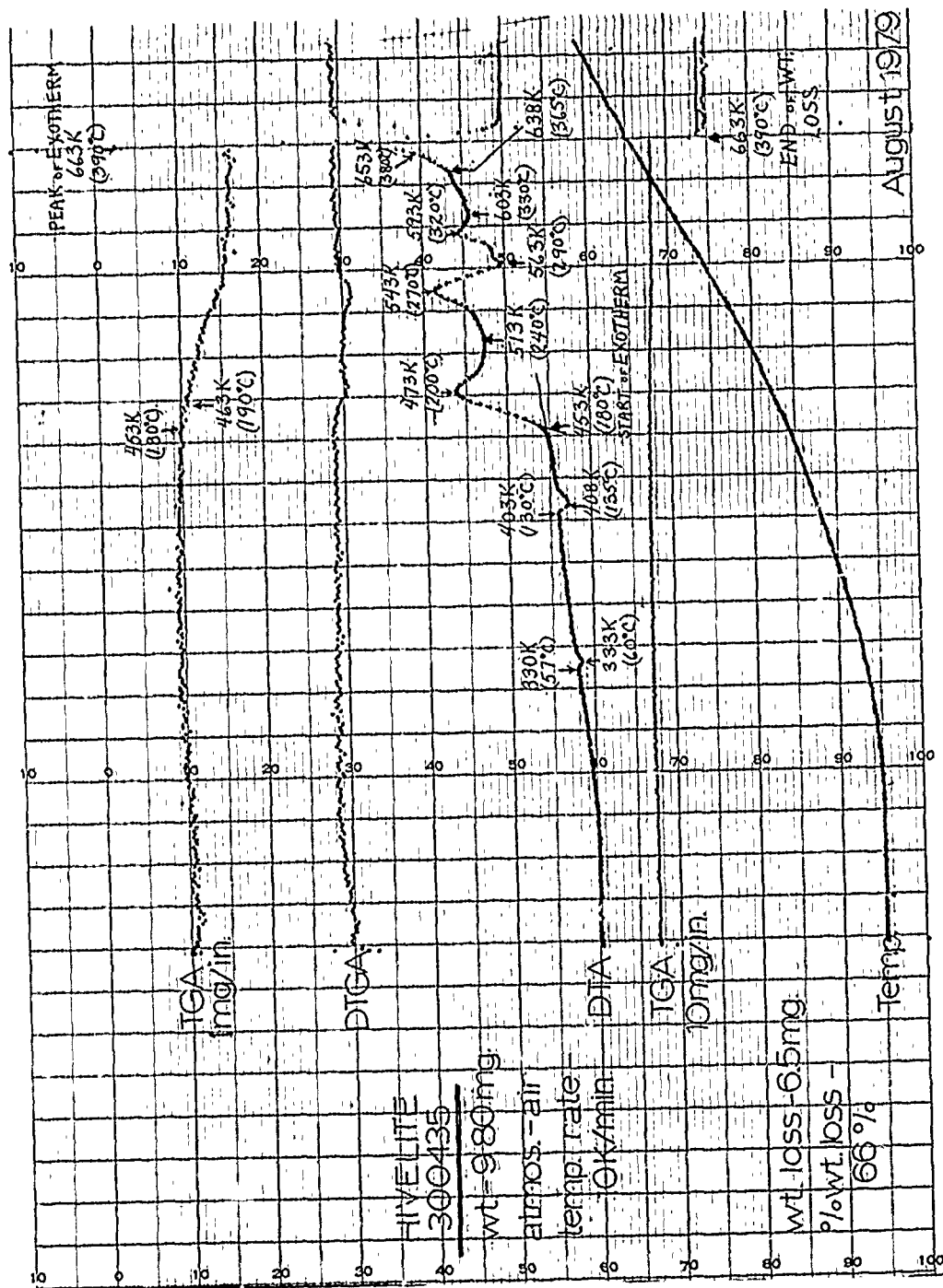
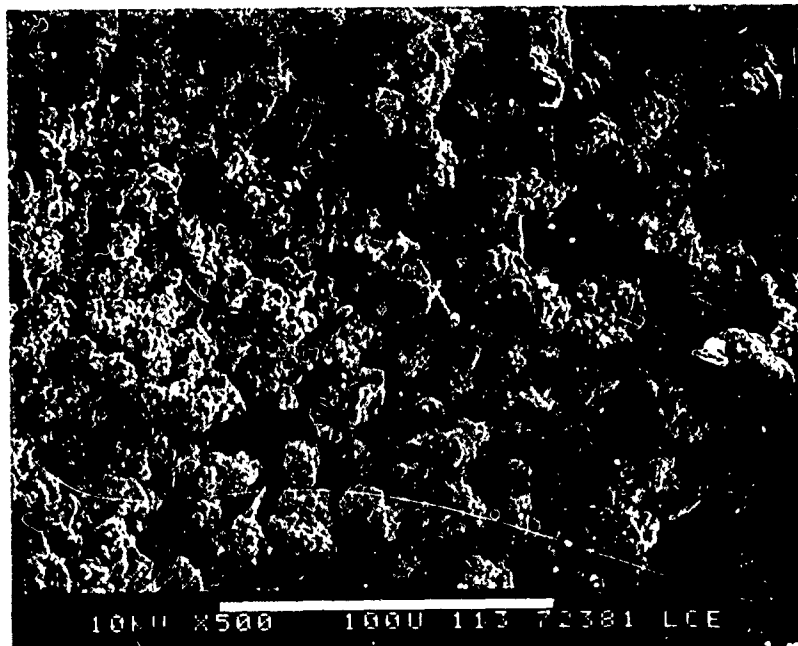


Figure 3. DTA/TGA thermograms of HIVE LITE 300435

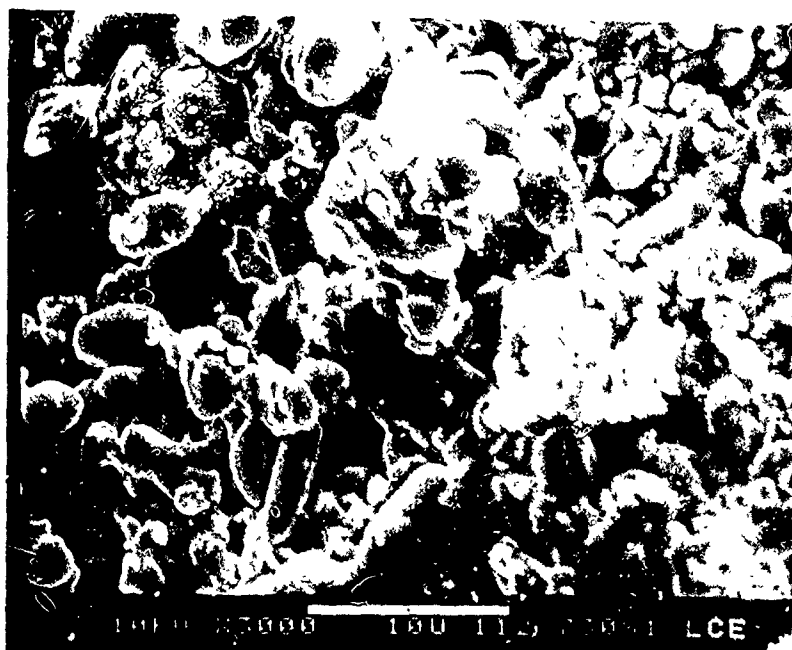


HIVE LITE



HIVE LITE ANNEALED

Figure 4. SEM photos of HIVE LITE 300511 before and after heating (500 X)



HIVELITE ANNEALED



HIVELITE

Figure 5. SEM photos of HIVELITE 300511 before and after heating (3000 X)



HIVELITE ANNEALED



HIVELITE

Figure 5. (cont)

CLOSED BOMB DATA
HIVELITE 300511
+21°C

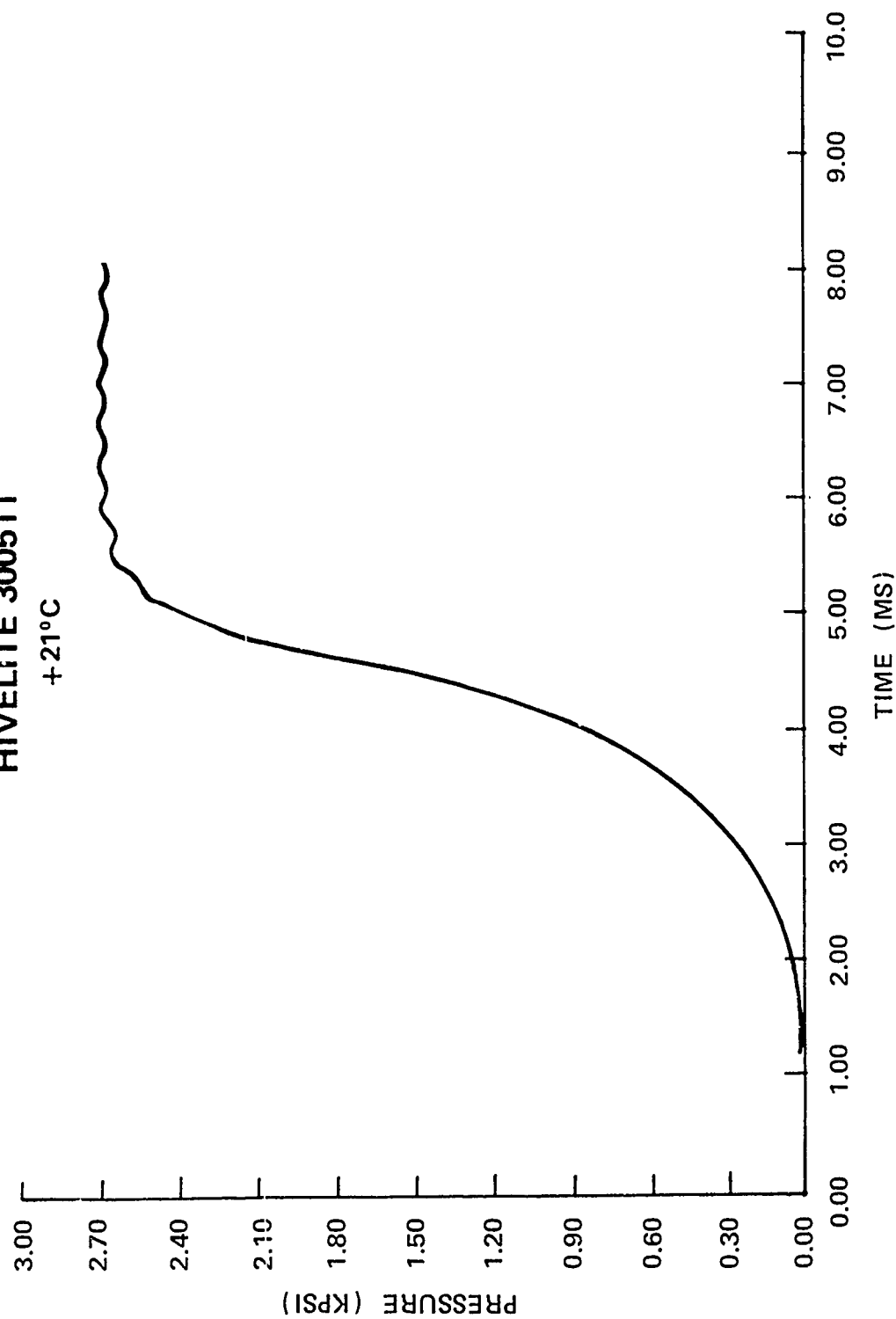


Figure 6. Pressure-time data for HIVELITE 300511 at 294 K (21°C)

CLOSED BOMB DATA
HIVELITE 300511

-51°C

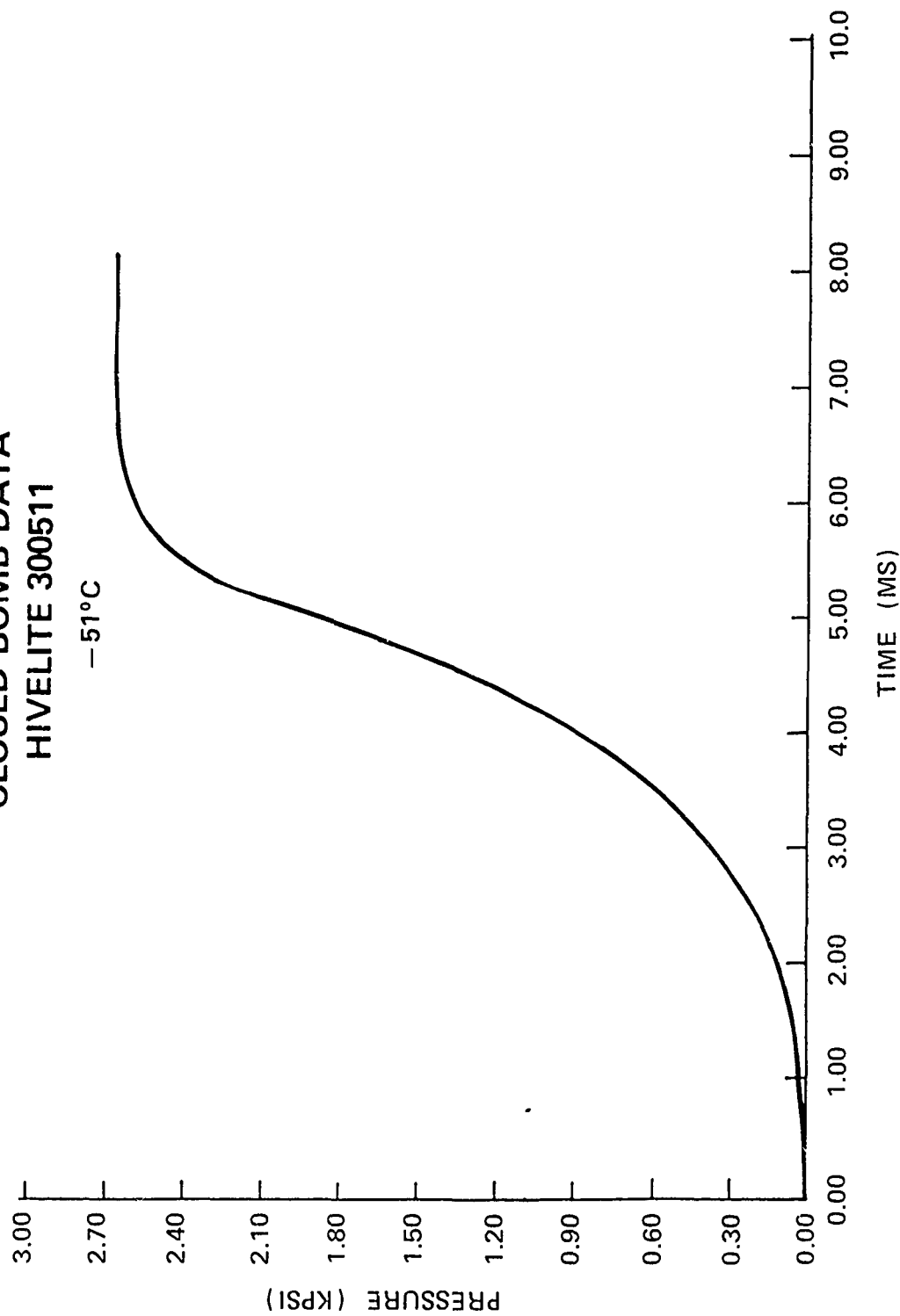


Figure 7. Pressure-time data for HIVELITE 300511 at 222 K (-51°C)

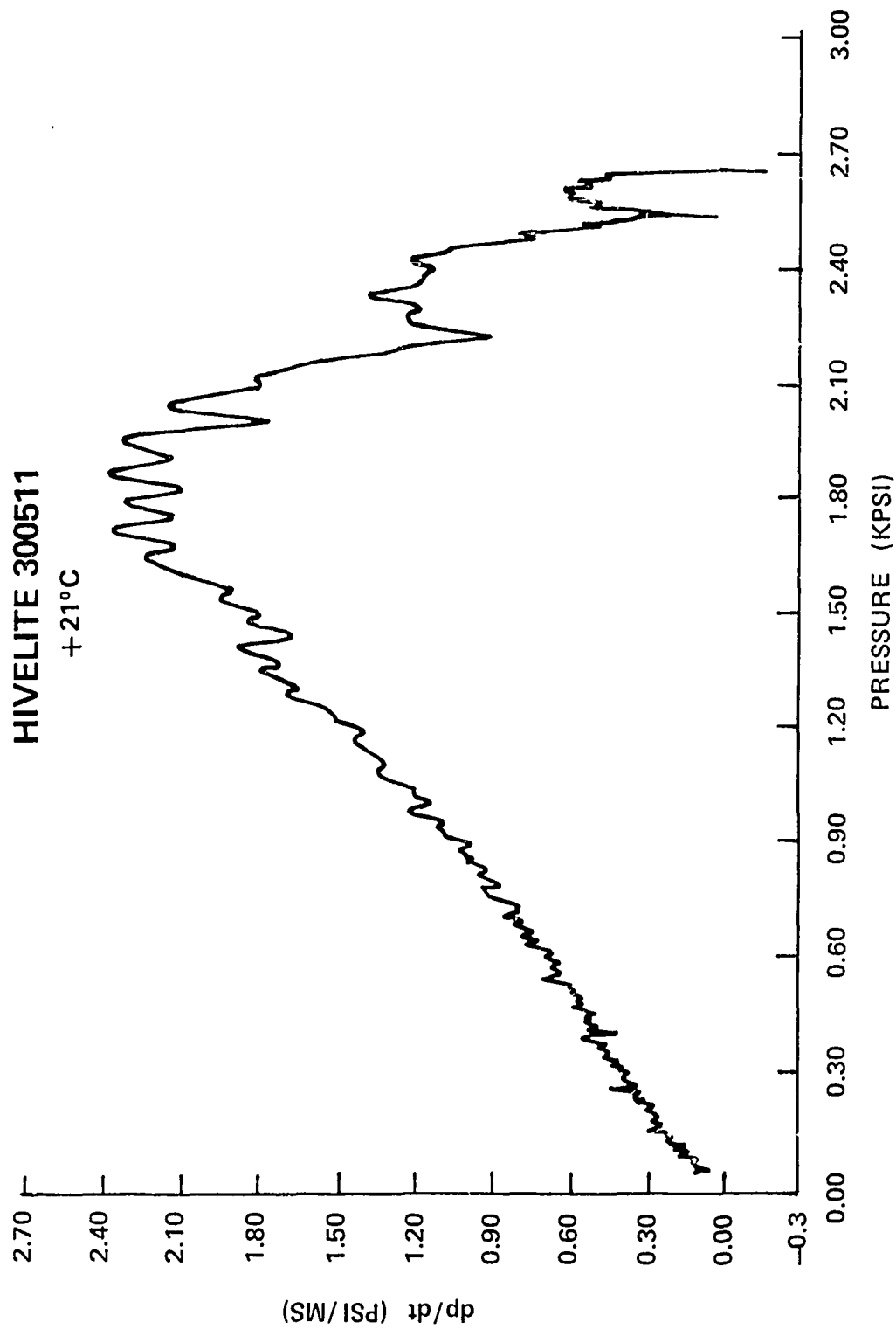


Figure 8. dp/dt versus P curve for HIVELITE 300511 at 294 K (+21°C) (Test 2)

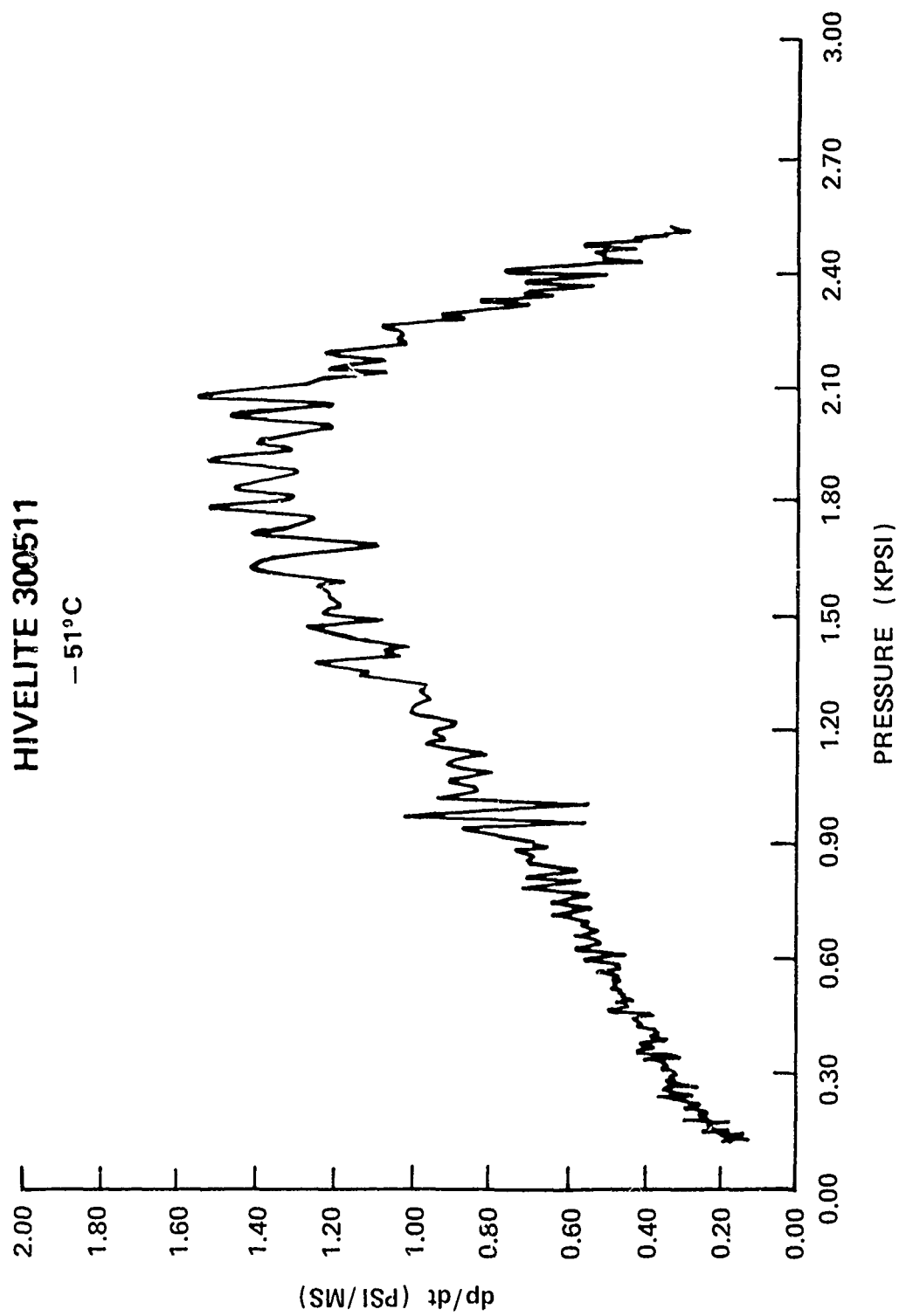


Figure 9. dp/dt versus P for HIVELITE 300511 at 222 K (-51°C) (Test 4)

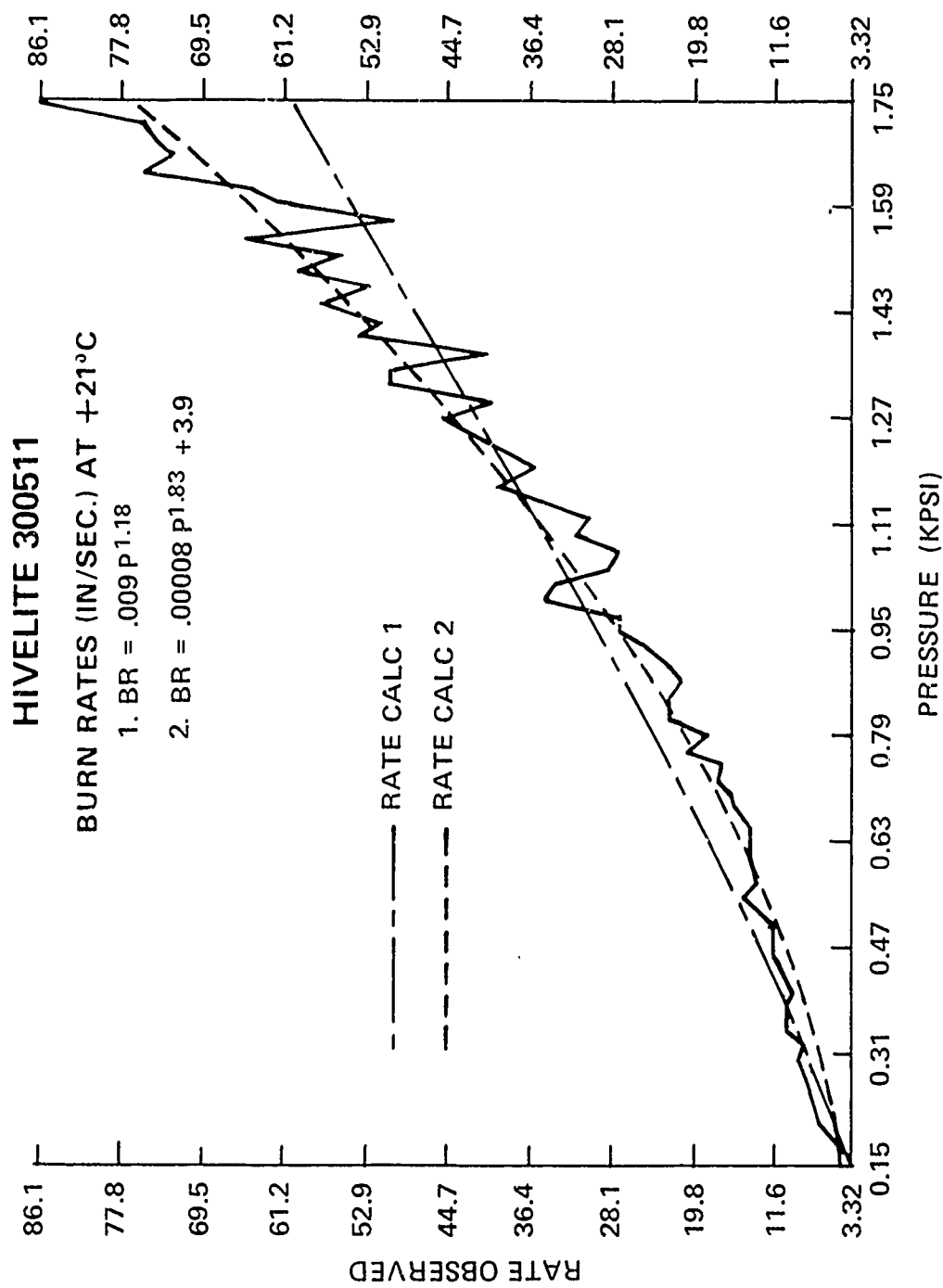


Figure 10. Composite burning rate for HIVELITE 300511 at 294 K (21°C)

HIVELITE 300511

BURN RATES (IN/SEC.) AT -51°C

1. BR = .0114 P^{1.10}

2. BR = .0001 P^{1.71} + 4.2

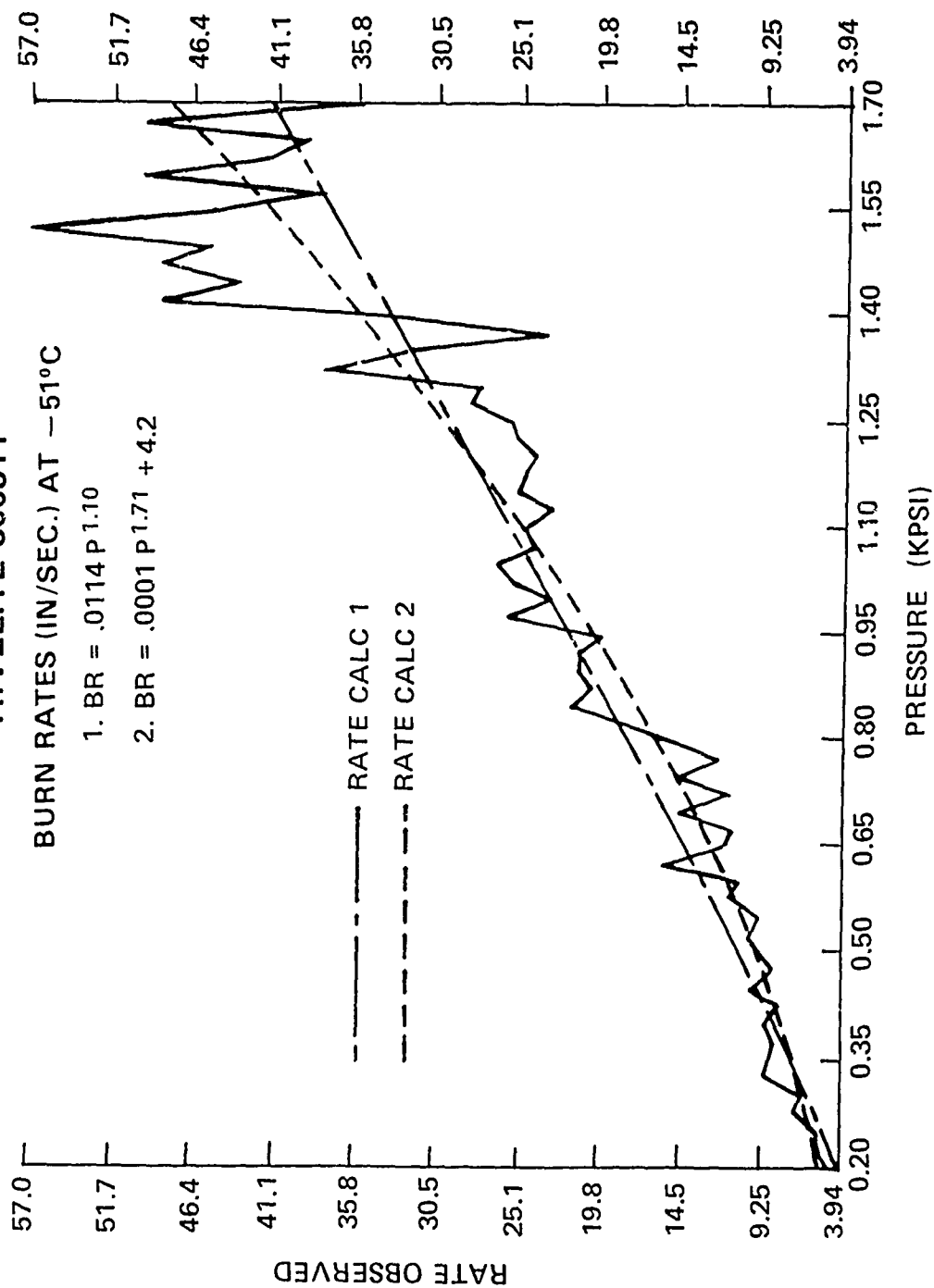
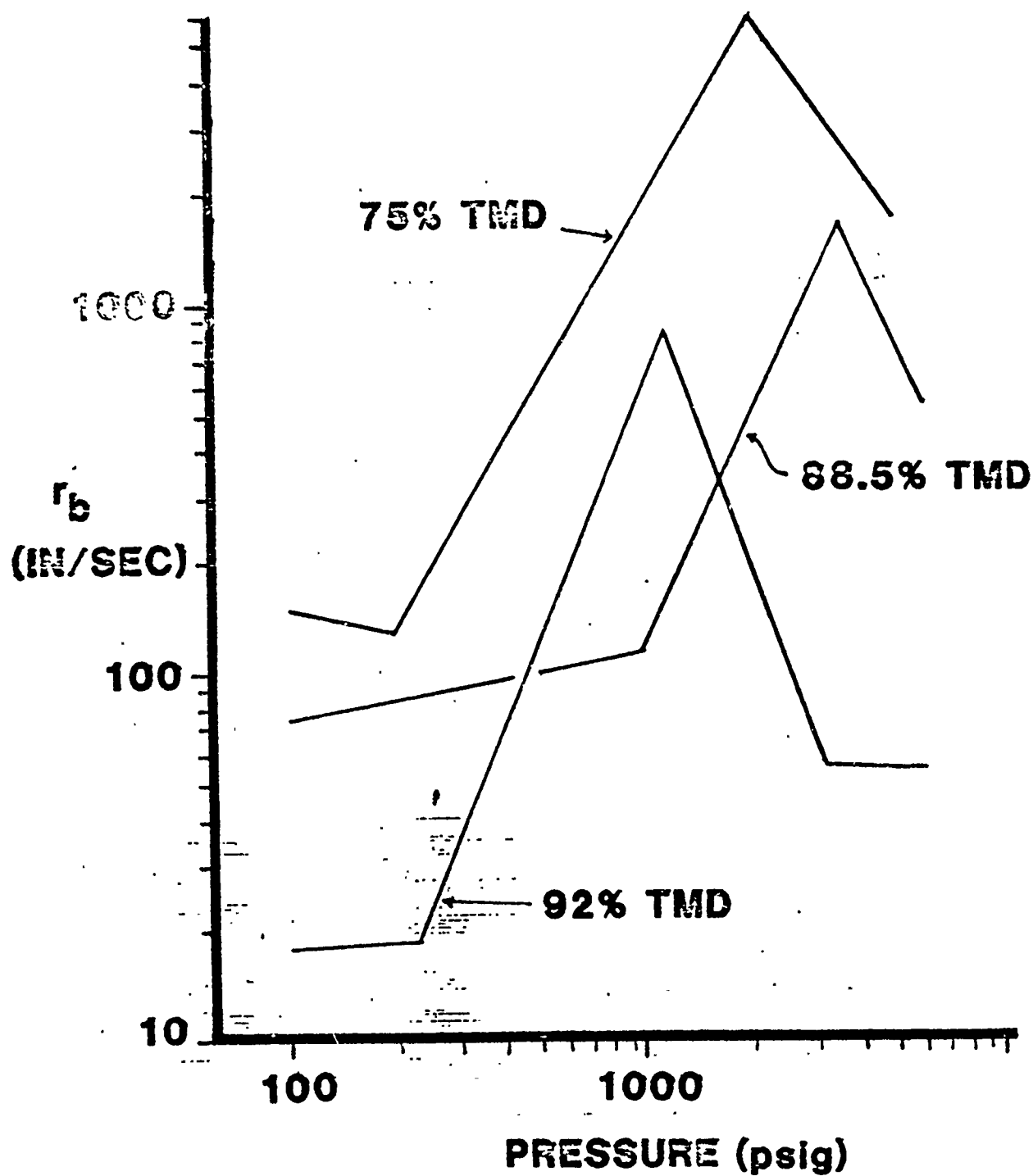


Figure 11. Composite burning rate for HIVELITE 300511 at 222 K (-51°C)



Series 1 & 3

Figure 12. TMeS closed bomb data for HIVEHITE 300511

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